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Research Note

Extraction and Characterization of Natural Fibres from Plantain (*Musa paradisiaca*) Stalk Wastes

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A B S T R A C T

Plantain stalks obtained from solid waste stream of Ganmo market in Ilorin was used in this study. Natural fibres extraction from waste plantain stalk was achieved using biological retting methods. The natural fibre was rented from the waste stalk after 24 days of soaking in water. The extracted fibres were exposed to 2, 4 and 6% alkali solution (NaOH) treatment for two hours, washed and dried in the oven for 7 hours. Elemental analysis of raw plantain fibres showed the presence of elements like Indium, Potassium, Silicon and Calcium among others. Tensile strength analysis of the fibres, for single fibre strands showed that the 2% treated fibre showed distinctly promising potential with the highest tensile characteristics of young modulus, stress at break and force at peak of 52864.366N/mm^2, 5398.536N/mm^2 and 2.650N, respectively. Evaluation of the chemical composition of plantain by FTIR spectroscopy indicated that treatment of natural fibres using NaOH beyond 2% have a negative impact on the plantain fibre properties. Through alkali exposure, the fibre configuration presents small variations in composition. It is consequently apparent that alkali treatment with concentration of less than 2% NaOH is sufficient to remove hemicelluloses and to obtain the optimum tensile effect.

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INTRODUCTION

Increased consumption of energy and resources worldwide has yielded positive yet unsustainable development. This gives rise to the need for a more sustainable use of resources and reduction in energy consumption. Lignocellulosic fibres which are major renewable resources derived from structural plants pose fulfillment for this need. Natural fibres are plants composed of cellulose and lignin. Natural fibres are not only renewable and biodegradable but also possess characteristic advantages over conventional fibres such as sustainable manufacturing process, improved modulus, improved strength, lightweight and low cost [1-3].

Natural fibres also have disadvantages, some of which include: poor wettability, high moisture absorption and non-compatibility with matrices. The use of chemical and physical treatments for surface modification of fibres has been discovered as a way to overcome these challenges and improve natural fibre properties [4].

Plantain fibre is a bast lignocellulosic fibre which can be extracted from the pseudo-stem of plantain after the utilization of the fruits and leaves [5, 6] Bast fibres are fibres consisting of lignin, hemicellulose and cellulose in various proportions as well as minerals, wax, water soluble compounds and pectin which serves as a glue to hold the fibres together as bundles. The pectin, gum and other mucilaginous substances contained in the bundles are decomposed during retting which aids in the extraction of the fibre.

Alkali treatment also known as mercerization is a chemical process of immersing natural fibres in a relatively concentrated aqueous solution of strong base to create sufficient swelling by the removal of hemicelluloses, lignin, waxy materials and impurities on the surface of the fibre cell wall. The use of alkali treatment of natural fibre also improves the tensile strength, fibre wetting by fibrillation, fibre-matrix adhesion due to the removal of both natural and artificial impurities, and oils covering the external surface of the fibre's cell wall. Alkaline solution depolymerizes the native cellulose structure and exposes short length crystallites [7]. Based on literature [8-10], alkaline treatment of fibres not only depolymerizes cellulose; but also increase in surface roughness of the fibre which in turn causes a higher extension of hydrogen bonds at the fibre-matrix interface.

The process and methods of extracting plantain fibre have not been fully developed. Researchers have suggested that the

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fluid or slurry content could be used for potential drug development because of its therapeutic value [11, 12]; while, the solid-fibre content could be further developed for fibrillated and reinforcing purposes [13, 14]. With the purpose of eliminating the manual methods and to increase the quality and improve the processing time, Oreko et al. [15] paid attention to the mechanical design and development of plantain fibre extraction machine in an attempt to achieve the automation of plantain fibre extraction. However, they recommended that further work is needed to achieve optimal performance of the plantain fibre extraction machine at low cost.

Waste conversion into wealth is of paramount interest to governments, researchers and scientists across developing countries. In Nigeria, the cultivation and utilization of plantain generates a large amount of agro-residues/ wastes [16]. Fibres extracted from the stem, pseudo-stem and empty fruit bunch of plantain can be used as reinforcement in polymer composites which has applications in structural engineering. The aim of this work is to obtain extract, treat and characterize raw and treated plantain fibres for tensile, Fourier Transform Infrared Spectroscopy FTIR and X-Ray Fluorescence (XRF) analyses.

MATERIALS AND METHODS

Plantain pseudo-stems were collected from the solid waste stream of Ganmo market in Ilorin. The plantain fibre was extracted using biological (water) retting methods as described by Sisti et al.[17]. The extracted fibres were dried at room temperature for about 3 days (72 hours). Details of the treatment fibers for defining categories list out in Table 1.

X-Ray fluorescence

Raw plantain fibres were analyzed for elemental and oxide composition analyses were carried out using X-Ray Fluorescence (XRF). X-ray fluorescence (XRF) Nitron 3000 was used for this analysis. The machine was powered on and was allowed to stabilize for 5 minutes after initialization. The method used for the analysis was Cu-Zn method which normally detect large amount of elements and sesquioxides due to its intensity. The sample was placed on the sample holder while the ray point was placed over it and the ray button was pressed in order to log data being observed. The data were collected in triplicates and this automatically takes the average.

Chemical (alkaline) treatment of plantain fibre

The extracted fibres of plantain were immersed in 2, 4 and 6% sodium hydroxide solution (wt%) at room temperature for about 4 hours and washed with distilled water to remove residual alkaline solution. The fibre samples were identified with the sample codes NT, 2T, 4T and 6T, respectively.

TABLE 1. Chemical treatment for defined categories

Categories	Chemical Treatment
NT	Untreated Plantain Fibre
2T	2% sodium hydroxide solution
4T	4% sodium hydroxide solution
6T	6% sodium hydroxide solution

Fibre tensile test

The tensile tests of fibrous polymers are generally performed using strands of fibers involved. The dimension of the tensile test sample was as per ASTM D3822 standard [18] test method for fibre properties. The length of the fibre strands used is 150 mm. The tensile test was performed in a universal testing machine (model UTM: M500-50). The tests were performed with a crosshead speed of 0.5 mm/min. For each test fibre strands of four samples were tested and the average value was taken for analysis. The analysis was carried out for single fibre strands.

Fourier transform infrared spectroscopy (FTIR) analysis

The FTIR analysis is an important indicative on the possibility of fibre integration with the polymer matrices. FTIR Spectroscopy was carried out on the treated and untreated plantain fibre samples to qualitatively identify the constituents of the fibre. The analysis was carried out using Thermo Scientific Nicolet iS5-iD1 machine for FTIR analysis in the Chemistry Department of University of Ilorin.

RESULTS AND DISCUSSION

Elemental composition of plantain fiber

The elemental composition of raw plantain fibres is presented in Table 2. The data showed that the five highest inorganic constituents of the plantain fibre were Indium, Silicon, Potassium, Calcium, and Aluminum in descending order. These elements and others present in smaller concentrations underpin the use of the extracted plantain fibres in diverse engineering applications. For example, if present in extracted quantities, indium and silicon are useful in the manufacture of electronic devices such as transistors, insulators, rectifies and chips among others. On its own, Indium oxide can also be doped with Tin oxide to manufacture thin conductive transparent films for use in photovoltaics. Previous works had confirmed the similar use of bio-extracted silicon and indium [19, 20].

TABLE 2. Elemental composition of plantain fibre

Element Name	Element Symbol	Percentage Composition (%)
Indium	In	35.01
Potassium	K	25.05
Silicon	Si	13.87
Calcium	Ca	7.76
Aluminium	Al	4.00
Chlorine	Cl	3.74
Sulphur	S	2.06
Iron	Fe	1.04
Titanium	Ti	0.19
Copper	Cu	0.18
Manganese	Mn	0.09
Arsenic	As	0.009
Lead	Pb	0.008
Barium	Ba	0.004

Plantain fibre can also be reprocessed for use in production of fertiliser because of the potassium and silicon content of the fibre [21]. Potassium can also be extracted from plantain fibre for industrial and health applications such as blood pressure reduction [22]. Plantain fibres can also be used in the production of Yarn, rope, high quality textiles and auto body works [16, 23, 24].

Tensile properties of plantain fiber

Changes in the mechanical properties obtained from the tensile analysis of Plantain Fibre with respect to treatment concentration, presented in Table 3, were analysed and presented in Figures 1 and 2.

Table 3 shows clearly that the 2% treated fibre (2T) exhibited the highest tensile characteristics of young modulus of 52.9GPa with tensile strength and force at peak of 5398.5MPa and 2.650N, respectively. The tensile strength of the fibre increases with alkaline treatment from 2084MPa to 5398.5MPa for a 2% treatment concentration which is about 160% increase in the property. However, further increment in the concentration of sodium hydroxide, (NaOH) solution yields in a reduction in the tensile stress of the fibre. This implies that the overtreatment of plantain fibre beyond 2% NaOH concentration for a 4-hour immersion time will have a negative impact on the base properties of the fibre.

Figures 1 and 2 show that the highest values of young modulus and tensile strength are attained at a treatment concentration of 2%. The curves showed that the mechanical properties were directly proportional to treatment concentration until it attained the highest value at 2% then starts to decrease. It can be inferred from these graphs that the mechanical properties of plantain fibres are optimum at a treatment concentration of 2% NaOH solution.

The sampled raw plantain fibre was found to have a higher tensile strength of 2084.0Mpa compared to that analysed by Imoisili et al. [1] and Ihueze et al. [25] who obtained a fibre sample with Tensile strength of 489.5MPa and 536.2Mpa, respectively. However, Imoisili et al [1] observed an appreciable increase in tensile strength for alkaline concentrations up to 3%. This variation in optimal concentration for alkaline treatment may be due to difference in treatment process. Imoisili et al. [1] washed the fibre with acetic acid after washing with distilled water while the fibres analysed in this study were washed with distilled water only.

Recent studies have also shown that the higher concentrations of alkaline treatment of fibre could have a negative impact on the mechanical properties of the fibre. Zin et al. [26] observed that treatment of banana fibre with 6% NaOH solution for two hours immersion time resulted in the higher tensile strength compared to fibres treated with the same concentration for 4 hours. It was found and concluded that alkaline treatment of the fibre beyond 6% concentration or longer duration of 2-hour immersion time for 6% alkali concentration results in a deterioration of the mechanical properties of the fibre [26].

Cai et al. [9] also observed that the Young's modulus of the abaca fibres treated with 5% NaOH solution increased by 41%, whereas those treated with 10 and 15% NaOH solution decreased by 24 and 29%, respectively.

TABLE 3. Mechanical properties of treated and untreated plantain fibre

NaOH Solution (w/w) (%)	Young's Modulus (GPa)	Tensile Strength (MPa)	Strain (%)
0	26.98	2084.04	9.85
2	52.86	5398.54	10.02
4	23.68	1071.56	4.64
6	10.02	327.477	4.86

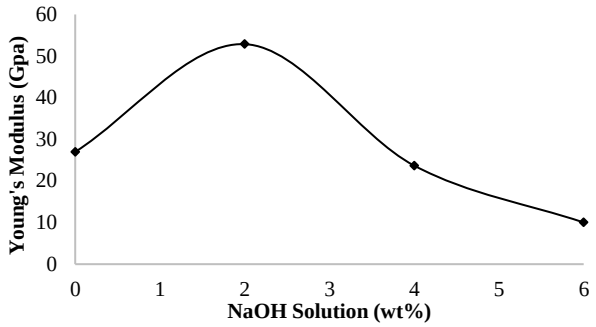

Figure 1. Effect of treatment concentration on the young modulus of the fibre

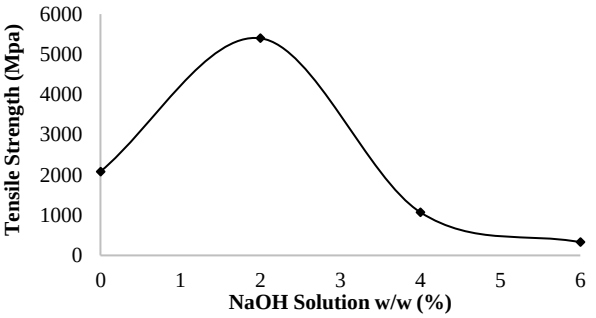

Figure 2. Effect of naoh concentration on the tensile strength of the fibre

FTIR spectroscopy of plantain fibers

The FTIR spectroscopy measures the intensity of light absorbed or emitted by the fibre at a particular wave length which corresponds to particular functional groups in the fibre. Figure 3 shows the FTIR spectra for plantain fibre samples, NT, 2T, 4T and 6T. Table 4 summarizes the characterisation spectra of untreated and treated plantain fibres as extracted from Figure 3.

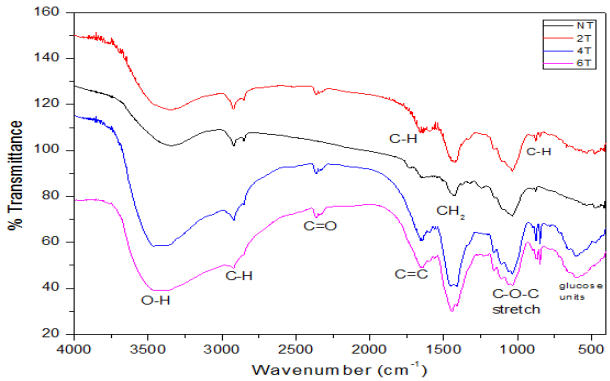

Figure 3. IR spectra of treated and non treated plantain fibres

TABLE 4. IR spectral analysis of treated and untreated plantain fibre

Functional Group	NT (cm^{-1})	2T (cm^{-1})	4T (cm^{-1})	6T (cm^{-1})	Possible Assignments
O-H stretching (H-bonded)	3344.59	3354.09	3464.91 3347.76	3452.24	Cellulose, Hemicellulose and Lignin
C-H stretching (aromatic)	2917.15 2850.66	2923.48 2850.66	2920.32	2923.48	Cellulose, Lignin, Carboxylic acids
C=C stretching (ester, aromatic ring, ketone)	1739.31 1650.66	1656.99	1659.89	1739.81 1650.66	Lignin, Hemicellulose
C-H bending (deformations)	1425.86	1425.86	1451.19 1406.86	1444.85 1410.03	Cellulose, Lignin
C-O	1242.22 1039.58	1033.25	1036.41	1232.72 1033.25	Lignin, hemicellulose
C-O-C stretch			1106.07	1159.89 1112.40	Cellulose
Glucose Units	878.10	881.27 249.60	878.10 852.77	871.77 846.44	β-glycosidic linkages between The monosaccharides

The broadband at 3344cm^{-1} for the raw fibre corresponds to hydrogen bonded –OH vibrational stretching of cellulose [27]. According to Monteiro et al. [28] this stretching is associated with –OH present in cellulose, lignin, hemicellulose and carboxylic acid. The absorption observed at 2917.15 and 2850.66cm^{-1} corresponds to asymmetric and symmetric stretching vibrations of the alkyl group in cellulose which is a characteristic of any natural fibre. The absorption band at 1739.13cm^{-1} and around 1650.66cm^{-1} may be attributed to stretching of the carbonyl (C=0) ester and carboxyl groups of hemicellulose and possibly fatty acids from lignin [29, 30].

The absorption at 1425.86cm^{-1} corresponds to aromatic ring vibrations while the small peaks between 1372.03 and 1242.22cm^{-1} characterises the C-H bending bond structure of the functional group of cellulose, hemicellulose and lignin while the peak at 1039.58cm^{-1} corresponds to a C–O– symmetric stretching vibration in cellulose, hemicellulose and minor contents of lignin [29, 31, 32]. The small absorption band at 878.10 cm^{-1} is attributed to the β-glucoside linkages between the sugar units in cellulose and hemicellulose [27].

The intensity of the peak around 3344cm^{-1} earlier noted for the raw fibre increases to about 3354cm^{-1} after treatment of the fibres, this increment may be due to the breakdown of lignin and part hydrogen bond of the OH group [25]. This characteristic peak of the hydrogen bonded (-OH) vibration indicates the presence of intermolecular hydrogen bonding which tends to shift to higher absorbance value in improved treated fibres [25]. It can be observed from presented data that only the 2% treated fibres have higher absorbance compared to NT fibres which implies that the 2T fibres are better than the untreated fibres while 4T and 6T fibres exhibited strength reduction.

The OH band becomes wider as the concentration of the alkali increases and this indicates the presence of a high concentration of cellulose. Also, the band 1739.31cm^{-1} which due to CH_3COO- and COOH functional group of hemicellulose observed in the raw fibre spectra are absent in the treated fibres. The disappearance of this characteristic vibrational stretching clearly indicates that the alkaline treatment has significantly removed the hemicellulose content [32]. The functional group analysis for further treatment of the fibres as shown in FTIR spectra, fibres are further illustrated reported in literature [9, 28, 29, 32].

The results of the FTIR analysis inferred that treatment with NaOH resulted in a gradual removal of hemicellulose and lignin which serve as binding components to the fibre. The treatment was also efficient in the exposition of lignin as indicated by the widening of the OH band. High amount of cellulose on fibre surfaces provides free hydroxyl groups that can react polymer groups in composite production.

CONCLUSION

Natural fibres were obtained readily at low cost from the waste plantain stalk via biological retting method. From the tensile test carried out values for the mechanical properties have been obtained for natural fibres under various alkali treatments, an understanding of how alkali treatment affects the extracted fibres is gained. The FTIR analysis also provides some insight into the important issue of understanding the structural behaviour of the extracted fibres under various alkali treatments. It can also be concluded that: the process of fibre alkalization has an effect on the base strength of natural fibre; there is an optimum process condition of fibre performance if the composite material development is to be maximized. The fibres could be used to develop fibre reinforced plastic composite components in use in engineering applications. Plantain fibre can also be reprocessed for use in the production of fertilizer because of the potassium and silicon content of the fibre as observed from XRF analysis. Further works are needed on plantain fibre morphological studies for more explicit classification and application.

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Persian Abstract

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چکیده

در این تحقیق از ساقه‌های چنار به دست آمده از جریان زباله جامد در بازار گانمو در ایلورین استفاده شد. استخراج الیاف طبیعی از ساقه درخت چنار با استفاده از روش‌های برداشت بیولوژیکی انجام شد. فیبر طبیعی پس از ۲۴ روز خیساندن در آب از ساقه زباله حاصل شد. الیاف استخراج‌شده به مدت دو ساعت در معرض محلول قلیایی ۲، ۴ و ۶ درصد (NaOH) قرار گرفتند و به مدت ۷ ساعت در فر قرار گرفته‌اند و خشک شدند. تجزیه و تحلیل مقدماتی الیاف چنار خام گیاهان حضور عناصری مانند ایندیوم، پتاسیم، سیلیکون و کلسیم را در بین سایرین نشان داد. تجزیه و تحلیل استحکام کششی الیاف، برای رشته‌های فیبر منفرد نشان داد که فیبر تحت فراوری ۲٪ دارای پتانسیل بالایی با بالاترین ویژگی کششی مدول جوان، استرس در هنگام شکست و نیرو در پیک به ترتیب ۵۲۸۶۴/۳۶۶ N/mm^2، ۵۳۹۸/۵۳۶ N/mm^2، ۲/۶۵۰ N هستند. ارزیابی ترکیبات شیمیایی گیاه با استفاده از طیف سنجی FTIR نشان داد که تیمار الیاف طبیعی با استفاده از NaOH بیش از ۲٪ تأثیر منفی بر خصوصیات فیبر چنار دارد. از طریق قرار گرفتن در معرض قلیایی، پیکربندی فیبر تغییرات کمی در ترکیب ارائه می‌دهد. در نتیجه آشکار است که فراوری قلیایی با غلظت کمتر از ۲٪ NaOH برای از بین بردن همی‌سلولز و به دست آوردن اثر کششی مطلوب کافی است.